

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

IN THE APPLICATION OF: DIANE M. ARTMAN, ET AL.

DOCKET NO.: 3226-01

CUSTOMER NUMBER: 26645

SERIAL NO.: 10/554,481

EXAMINER: TAIWO OLADAPO

FILED: OCTOBER 24, 2005

GROUP ART UNIT: 1797

TITLE: DIESEL LUBRICANT LOW IN SULFUR AND PHOSPHORUS

Dated: January 5, 2009

Hon. Commissioner for Patents
P. O. Box 1450
Alexandria, VA 22313-1450

Sir:

DECLARATION UNDER 37 C.F.R. §1.131

We, DIANE M. ARTMAN, WILLIAM D. ABRAHAM, VIRGINIA A. CARRICK, AND JONATHAN S. VILARDO, declare as follows:

1. We are the applicants of the above-identified patent application and inventors of the subject matter described and claimed therein.

2. Prior to April 4, 2003 we completed (conceived and reduced to practice) the invention described in the above-identified application.

This is evidenced by a Technology Disclosure Report. Chemical identities and formulations of the compositions and processes of the present invention are provided in section 4 along with the results of the testing completed.

| | |
|---|---------------------------|
| I certify that this correspondence is being deposited with the United States Postal Service as <u>first class</u> <u>express mail</u> in an envelope addressed to Commissioner for Patents, POBox 1450, Alexandria VA 22313-1450 (EPA Mail) | |
| on <u>1-20-09</u> | By: <u>Nancy S. Dedek</u> |

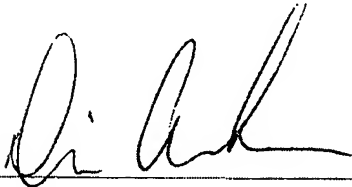
10/554,481

A photocopy of the document referred to above is attached. The document is dated prior to April 4, 2003 although the dates have been redacted. We have also redacted all internal material identifiers and codes, but have left generic material descriptions where provided.

Thus we had completed the conception and reduction to practice of the invention prior to April 4, 2003 as evidenced by the fact that a Technology Disclosure Report, including a description of testing completed, was previously completed and signed. This document was submitted and signed by each of the inventors listed in this application.

All the acts referred to above took place in this country.

We further declare that all statements herein made of our knowledge are true and all statements herein made on information and belief are believed to be true. We understand that willful false statements and the like are punishable by fine or imprisonment or both (18 U.S.C. 1001) and may jeopardize the validity of the application or any patent issuing thereon.



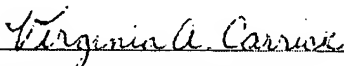
DIANE M. ARTMAN,

1-14-09 (date)



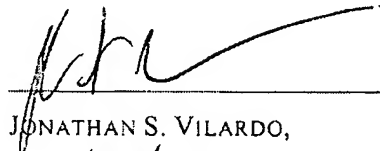
WILLIAM D. ABRAHAM,

1/9/09 (date)



VIRGINIA A. CARRICK,

1/9/09 (date)



JONATHAN S. VILARDO,

1/9/09 (date)

→ To: J. F. Munson/J. F. Pindar – IPLD (Wickliffe - Drop 022B)

TECHNOLOGY DISCLOSURE REPORT

IPLD use

T 1351

From:

| | | | | |
|-----------|-------------------------|------|--------------------|----------|
| Submitter | <i>Virginia Carrick</i> | | Virginia Carrick | VACA |
| | Signature | Date | Print or Type Name | Initials |
| Submitter | <i>Jon Vilardo</i> | | Jon Vilardo | JVIL |
| | Signature | Date | Print or Type Name | Initials |
| Submitter | <i>Bill Abraham</i> | | Bill Abraham | WDA |
| | Signature | Date | Print or Type Name | Initials |
| Submitter | <i>Diane Artman</i> | | Diane Artman | DMA |
| | Signature | Date | Print or Type Name | Initials |

The submitter need not believe that he or she is an inventor in order to submit a report. The designation of inventors is a legal question and determination will be made by IPLD.

Carefully review the report before submitting. Any questions should go to IPLD.

Information provided by submitter(s) must be correct or reasonably believed to be correct to the best of their knowledge. Any statements in this report are preliminary and subject to legal or technical correction.

Use additional pages as necessary to complete each portion of the report, with reference to the number heading of the section on each added sheet. Attach and date the extra sheets, and record in section 11 the number of sheets added. If no sheets are added, fill in "0."

Use standard chemical, electrical and mechanical terminology to describe the invention. To the extent known, code identities like LZ numbers should be placed after a named reactant, product, or intermediate.

The original of this disclosure should be **written in ink or typed/printed** and sent to IPLD as soon as possible. Additionally an electronic version would be of great assistance. The signed original hard copy should always be sent to IPLD for safekeeping.

When a TDR is elevated to a case (i.e. approved for drafting a patent application), the submitter should supply: (1) any prior art that he or she is aware of. It is not necessary to conduct a prior art search, but the results of any such searches already in hand are very helpful. (2) if the TDR is not already up to date in detail, an update of sections 5 and 6 with an appendix, providing a further detailed description of the invention, including data that may be used in the patent application. This should be submitted as an electronic document with the data, preferably in tabular form appropriate for the patent application. Code identities should be provided in parentheses next to the chemical names. The code identities will be removed from the patent application, so complete names suitable for external communication are necessary.

Alternatively, if you want to try your hand at creating a draft provisional patent application, a template is available at the end of this form. The hyperlink may or may not work depending on your location. Submitting such a draft would be most helpful any time, but especially for urgent cases.

1. Title brief description of the disclosure (e.g. compound name, gas additive for valve seat recession.)

Sulfurized antiwear component (sulfurized 4-carbobutoxy cyclohexene treated with TPP) giving good wear performance in a low phosphorus, low sulfur crankcase oil.

2. Background of the Invention (Publications and Patents) Attach copies and a list of known patents and papers relating to problem solved. No search need be conducted.

Projected global engine emission standards for the 2002-2010 time period, namely Euro IV, Euro V and PC-10, will require revolutionary changes in the formulation of future HD crankcase oils. Engine and vehicle manufacturers are beginning to target or to imply a need for lubricant formulation changes leading to significant reductions in sulfur and phosphorus levels in future engine oils. The lower levels of sulfur and phosphorus will have a serious impact on engine oil performance, especially in the area of wear protection and specifically high sliding wear conditions. Recent screen and engine testing has shown that a sulfurized antiwear component can offer wear performance benefits in low phosphorus, low sulfur diesel oil formulations.

3. Problem Solved Give a general description of the problem solved (e.g., unleaded gas contributes to valve seat recession in engine. Na alkyl benzene sulfonates in gasoline were found to diminish recession.)

Reduction of sulfur and phosphorus levels in future diesel oil formulations will necessitate the reduction of zinc dialkyldithiophosphate (ZDP) in the formulation. The reduction of ZDP will lead to much reduced antiwear protection of the oil. By using a sulfurized antiwear component in a low phosphorus/low sulfur diesel oil, injector adjuster screw wear (as measured by the short M11 test) can be significantly reduced.

4. Summary of the Invention (Compositions, Process or Device Descriptions).

Chemical terminology must be used for chemical inventions. Code identities or numbers should be added in parentheses if known, but must not be used alone. Metric (SI) units must be used throughout, optionally followed by conventional units in parentheses. If the invention is better described by flow diagrams or figures, these can be attached as numbered appendix pages and their relevance described here. Respond to as many of the following bullets as applicable.

- Composition - comprising (a), (b), (c) and (d) etc. (show chemical structures if appropriate). Distinguish between those components that appear to be important in solving the problem, versus those which are conventionally present (diluent, anti-foams, etc.)
 - ChemDraw structures in the TDRs submitted electronically may be useful in the patent
- Method or Process - comprising the steps (a), (b), (c) and (d) etc.
- Device - comprising the following parts: [list the parts] {provide a drawing}

Baseline 1

Baseline 1 is [REDACTED]

CHEVRON RLOP BASE SAE 15W-40 [REDACTED] (90%W [REDACTED]) + [REDACTED] (10%W [REDACTED]) + 8.2%W [REDACTED] + 0.2%W [REDACTED]
 13.0%W concentrate:
 7.2%W [REDACTED]
 0.389%W dil oil
 1.15%W [REDACTED] (secondary zinc dithiophosphate)
 1.20%W [REDACTED] (high TBN Ca sulfonate)
 0.90%W [REDACTED] (low TBN Ca sulfonate)
 0.76%W [REDACTED] (90 TBN Ca phenate)
 0.87%W [REDACTED] (255 TBN Ca phenate)
 0.5%W [REDACTED] (sulfurized olefin)
 0.031%W [REDACTED] (DMTD derivative)
 100 PPM [REDACTED] (silicone defoamer)

Baseline 2

Baseline 2 is [REDACTED] with the Ca phenates replaced with Mg saligenin (zero-S detergent) on an equal substrate basis and rebalancing the sulfonates to give equal sulfonate substrate and equal ash to Baseline 1. [REDACTED] was replaced with zero-S antioxidants ([REDACTED] and [REDACTED]) and the ZDP was reduced to deliver 0.05%P.

CHEVRON RLOP BASE SAE 15W-40 [REDACTED] (90%W [REDACTED]) + [REDACTED] (10%W [REDACTED]) + 8.2%W [REDACTED] + 0.2%W [REDACTED]
 12.46%W concentrate:
 7.2%W [REDACTED]
 0.389%W DIL OIL
 0.5%W [REDACTED] (secondary zinc dithiophosphate)
 2.05%W [REDACTED] (high TBN Ca sulfonate)

0.38%W (low TBN Ca sulfonate)
1.31% (Mg saligenin)
0.4%W (hindered phenol)
0.2%W (amine)
0.031%W (DMTD derivative)
100 PPM (silicone defoamer)

Invention Formulation

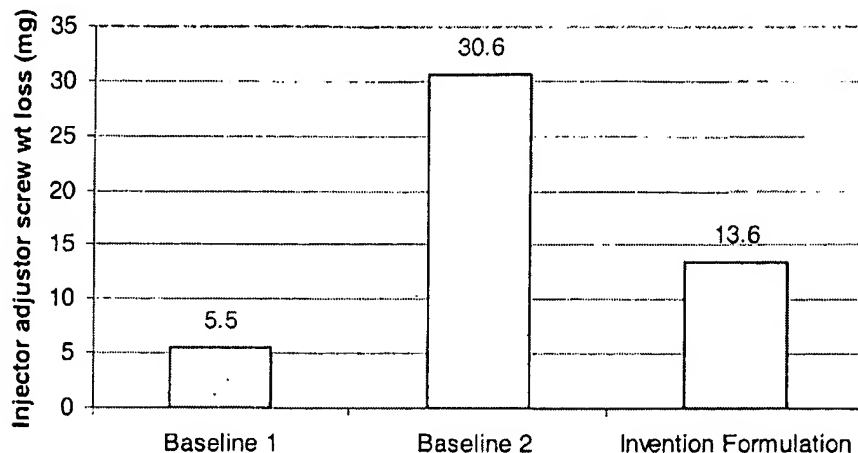
The formulation of this invention is Baseline 2 with addition of 0.5%

CHEVRON RLOP BASE SAE 15W-40 () (90%W) + () (10%W) + 8.2%W + 0.2%W
12.96%W concentrate:
7.2%W
0.389%W DIL OIL
0.5%W (secondary zinc dithiophosphate)
2.05%W (high TBN Ca sulfonate)
0.38%W (low TBN Ca sulfonate)
1.31% (Mg saligenin)
0.4%W (hindered phenol)
0.2%W (amine)
0.031%W (DMTD derivative)
0.50% (sulfurized antiwear component)
100 PPM (silicone defoamer)

The table below shows the detailed breakdown of the formulations along with P and S levels.

| | Baseline 1 | Baseline 2 | Invention Formulation |
|----------------------|------------|------------|-----------------------|
| Conc S010-0120-99- | 01 | 03 | 961 |
| Blends S010-0120-99- | 04 | 06 | 962 |
| | | | |
| RLOP [REDACTED]) | 90 | 90 | 90 |
| RLOP [REDACTED]) | 10 | 10 | 10 |
| [REDACTED] | 8.2 | 8.2 | 8.2 |
| [REDACTED] | 0.2 | 0.2 | 0.2 |
| | | | |
| [REDACTED] | 7.20 | 7.2 | 7.2 |
| [REDACTED] | 0.90 | 0.38 | 0.38 |
| [REDACTED] | 1.20 | 2.05 | 2.05 |
| [REDACTED] | 0.76 | | |
| [REDACTED] | 0.87 | | |
| [REDACTED] | 1.15 | 0.5 | 0.5 |
| [REDACTED] | 0.031 | 0.031 | 0.031 |
| dil oil | 0.389 | 0.389 | 0.389 |
| [REDACTED] | 0.50 | | |
| [REDACTED] | | 1.31 | 1.31 |
| [REDACTED] | | 0.4 | 0.4 |
| [REDACTED] | | 0.2 | 0.2 |
| [REDACTED] | | | 0.5 |
| [REDACTED] | 0.01 | 0.01 | 0.01 |
| | | | |
| Treat | 13 | 12.46 | 12.96 |
| | | | |
| %P | 0.116 | 0.052 | 0.053 |
| %S | 0.429 | 0.192 | 0.255 |

Testing of the compositions described above was carried out in the short M11 and gave the following results:



- The results show that:

The results show that the formulation containing [REDACTED] gives reduced injector adjuster screw weight loss as measured in the short M11 test.

Short Cummins M11 Engine Test - Another approach in obtaining equivalent wear performance information to a very expensive heavy duty diesel valve train wear (VTW) test such as the API CH-4 Cummins M11 (approx. \$55,000) is to develop an accelerated version of the engine test. Consequently, we have developed a "modified" M11 procedure which correlates reasonably well in terms of VTW to the API CH-4 Cummins M11.

The "modified" M11 procedure reduces the four stages (two 50 hour soot loading stages and two 50 hour wear stages) to two stages: one stage that generates 5.5% soot in a short time frame and then one stage that accelerates wear. This procedure is much shorter in length, permits a flush and fill approach, and costs only 25% of a full length CH-4 M11 engine test.

5. Detailed Description of the Invention. This section and the next (5. and 6.) may be abbreviated for the initial submission of the TDR, but they will eventually be needed in complete detail for filing a patent. Please respond to the bullets below as appropriate. Attach additional sheets as needed. (Using the attached template for a provisional patent application may be a simpler alternative.)

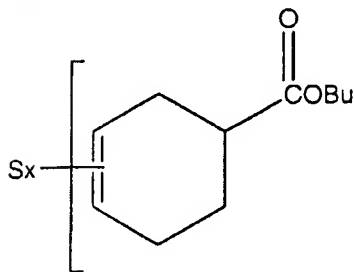
Calculations (%) are oil free.

- Component (a) is sulfurized antiwear agent. [for formulation composition inventions]
 - Specific examples of this type of component include [REDACTED]
[Provide preferred components]
 - The amounts of component (a) in the compositions of the present invention are generally 0.05-1.0%.
 - Preferably the amount of component (a) is 0.10-0.80%.
 - More preferably the amount of component (a) is 0.15-0.70%.

- Component (b) is zinc dithiophosphate.
 - Specific examples of this type of component include [REDACTED]
 - The amounts of component (b) in the compositions of the present invention are generally 0.02-0.09% P.
 - Preferably the amount of component (b) is 0.025-0.085% P.
 - More preferably the amount of component (b) is 0.03-0.08% P.
- Component (c) is succinimide dispersants.
 - Specific examples of this type of component include [REDACTED] and [REDACTED]
 - The amounts of component (c) in the compositions of the present invention are generally 1.5-9.0% (oil-free) total.
 - Preferably the amount of component (c) is 2.0-8.5% (oil-free) total.
 - More preferably the amount of component (c) is 2.5-8.0% (oil-free) total.
- Component (d) is phenolic and/or aminic antioxidant.
 - Specific examples of this type of component include [REDACTED] and [REDACTED]
 - The amounts of component (d) in the compositions of the present invention are generally 0.1-5.0% each.
 - Preferably the amount of component (d) is 0.15-4.5% each
 - More preferably the amount of component (d) is 0.2-4.0% each
- Component (e) is high TBN Ca sulfonate.
 - Specific examples of this type of component include [REDACTED] and [REDACTED]
 - The amounts of component (e) in the compositions of the present invention are generally 0.2-3.0% (oil-free).
 - Preferably the amount of component (e) is 0.25-2.5% (oil-free).
 - More preferably the amount of component (e) is 0.3-2.0% (oil-free).
- The particular relationship between the amounts and types of the above components is a sufficient amount of [REDACTED] in the presence of lower amounts of ZDP (e.g. invention formulation) provides adequate antiwear protection.
- The composition described above is prepared by normal blending procedures.
- The first step in carrying out the method of the invention is: [for method or process inventions]

6. Examples, Supporting Data, Test Results. For compositions supply one or more detailed recipes for making the compositions in question. Use chemical English. Include code identities like Lubrizol numbers in parentheses if known. Give supplier and the supplier's name for the chemical as well as the chemical name. Remember to account for diluent oil.

[REDACTED] is a 4-carbobutoxy cyclohexene treated with 3.5% TPP.



7. Use of Product or Process or Device. Give a brief discussion of one utility of the product or process or device (e.g., add Na C₂₄ alkyl benzene sulfonate to gasoline at 2 kg/1000L to control valve seat recession.) This is preferably supported by performance data/test results shown in sections 5 or 6.

8. Details of Experimental Use or Disclosures Outside of Lubrizol

Describe any samples that have left Lubrizol, to whom they went, when they left, whether a secrecy agreement was in place, and whether there was partial or full disclosure (of chemical identity, for example) outside of Lubrizol. This information should be updated and sent to IPLD when a case has been approved for drafting.

OS numbers

| (date) | (company) | (number) |
|--------|-----------|----------|
| | | |

9. Date product first made or process operated when & where; who observed the tests

S010-0120-99-04 (Baseline 1)

S010-0120-99-06 (Baseline 2)

S010-0120-99-962 (Invention Formulation)

[REDACTED]
[REDACTED]
[REDACTED]

10. People possibly involved in making the invention: [Include yourself if appropriate.]

Virginia Carrick
Jon Vilardo
Bill Abraham
Diane Artman

11. Witness and approval

Two Lubrizol employees who are not listed as a submitter must witness this disclosure. The witnesses, like the submitter(s), must read and completely understand the disclosure. The witnesses need not have seen the work conducted or have seen the submitter(s) sign the disclosure.

READ AND UNDERSTOOD BY ME: including _____ attached sheets

July A. Kocio
Witness

[REDACTED]
date

Don Bailey
Witness

[REDACTED]
date

APPROVED BY SUBMITTER'S SUPERVISOR:

By: Diane Artman

[REDACTED]
Date

By: James J. [Signature]

[REDACTED]
Date

Template for a Provisional Patent Application. If you are so inclined, you may create a draft provisional patent application as a means of communicating the detailed description of the invention. Detailed descriptions are needed when the TDR is elevated to a case, whether provisional or regular filing is planned. An electronic template is provided below. If sections 5 and 6 already contain an up-to-date detailed description of the invention, it may not be necessary to provide further information.

Click button for template:

